SENIOR THESIS

The Orientation of a Spinel Crystal From a Laue Photograph

by
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Introduction

A primary use of Laue photographs is for the orientation of single crystals. One method of determining the orientation of a crystal from a Laue photograph is by using stereographic projection. This was used to determine the orientation of a spinel from a transmission Laue photograph. The procedure is first to transfer the diffraction spots on the film to a stereographic projection, and then to locate the zone axes on the stereographic projection. Next the angles between the zone axes are measured and the axes are identified from these angles. With the zone axes identified it is then possible to locate all of the major zones on the stereographic projection. This method is primarily useful for isometric crystals like spinel. For nonisometric crystals it is necessary to know the cell dimensions of the crystal.

The Transmission Laue Method

The transmission Laue method is an x-ray diffraction technique that uses a single crystal placed between a film and an x-ray source. The x-ray beam passes through the crystal and is incident perpendicular to the film (see fig.1). The crystal does not move during the during the exposure of the photograph. The upper righthand corner of the film (as viewed from the crystal) is cut off to record its orientation with respect to the crystal. A diffraction maxima which gives rise to a spot on the film occurs when the condition $n\lambda = 2d\sin\theta$ is satisfied. Where $n=1,2,3,\ldots$, θ is the angle at which the x-ray beam is incident on a plane in the crystal, λ is the wavelenth of the x-rays, and d is the spacing between the diffracting planes. Since θ and λ are fixed it is necessary to use a continuous range of wavelenths to get spots from many planes in the crystal. An important feature of transmission Laue photographs is that the diffraction spots lie on ellipses. All of the spots on one ellipse represent planes in the crystal that belong to the same zone. A zone is a group of planes whose intersecting edges are parallel. The zone axis parallel to the intersection of these edges and perpendicular to the plane normals.

Determination of the Orientation of a Spinel Crystal by Stereographic Projection

1. Identification of the zones

The first step in determining the orientation of the spinel crystal is to identify the zones on the photograph. The zones can be identified because the spots falling on the same ellipse belong to the same zone. The five most obvious ellipses were labelled A,B,C,D and E (fig. 2). Five or more spots from each zone were transferred from each zone to the stereographic projection.

2. Transfer of spots to the stereographic projection

Each spot on a Laue photograph represents a set of parallel planes in the crystal. It is desirable to represent these spots on the stereographic projection as the poles normal to the planes in the crystal. This will allow the angles between the poles to be measured. The relation between the position of the spots on the film to the position of the poles on the projection is shown in figure 3. From this construction the following relations can be obtained

$$\text{film distance} = OS = D\tan 2\theta \qquad (1)$$

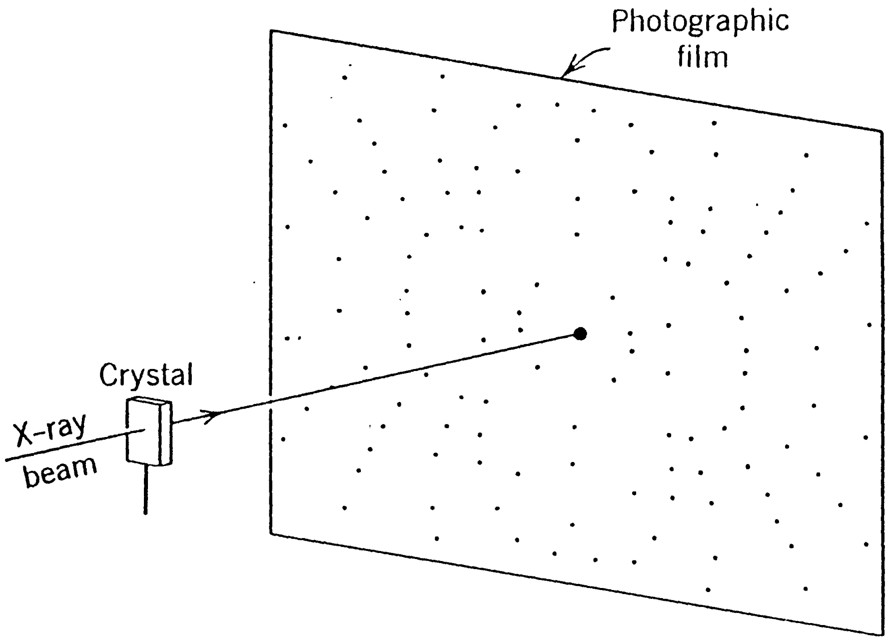
$$\text{projection distance} = PQ = R\tan(45-\theta/2) \qquad (2)$$

Where R is the radius of the stereographic projection and D is the crystal to film distance. The procedure used to transfer the spots from the film to the projection is as follows:

(a)Put a tack through the center of the Laue photograph, tape it to a light table and tape a piece of tracing paper over it. The photograph should be oriented so that the cut corner is at the upper left (as if the film is between the observer and crystal).

(b)Measure the distance on the film from the center of the photograph to a spot. This is the distance OS in formula (1).

(c)Calculate PQ from formulas (1) & (2)

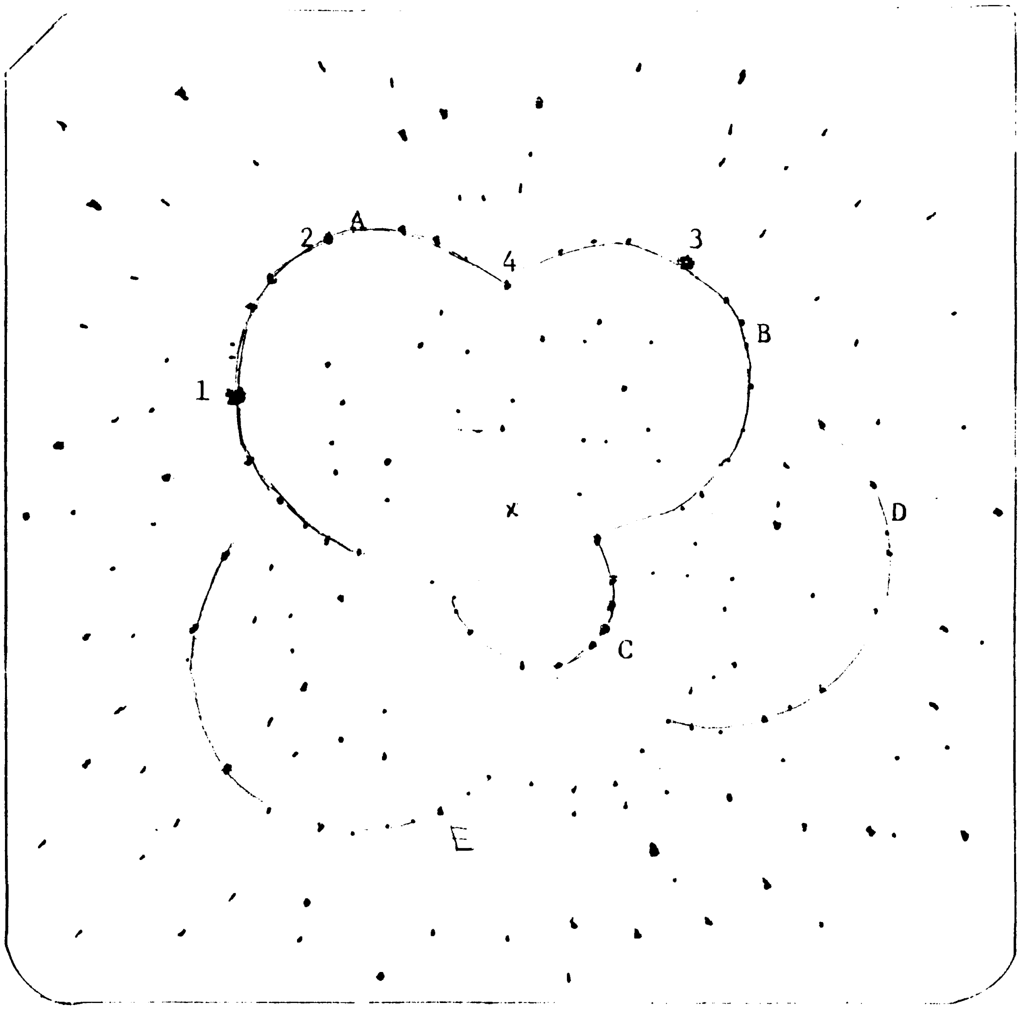


from Hurlbut and Klein p. 111

fig. 1

L A U E   P A T T E R N

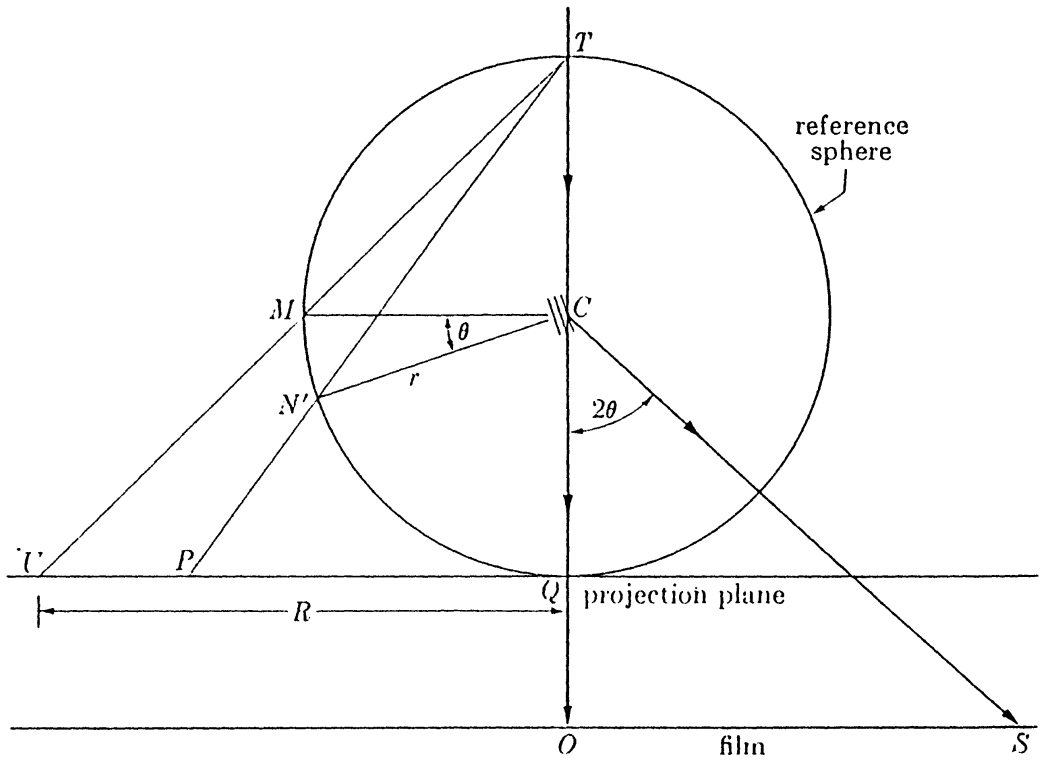
Spinel



Actual Size

copied from actual photo by R. Tettenhorst

fig. 2



from Cullity p. 234

(d)With an ordinary ruler in the same position as for the measurement of OS, mark a point on the projection a distance equal to PQ from the

center of the projection.

(e) Repeat (b)-(d) for all spots to be transferred to the stereographic projection.

3. Location of the zone axes on the stereographic projection

All of the planes that belong to to the same zone are parallel to the zone axis. This means that their normals are all perpendicular to the zone axis. Because of this the projected spots of one zone will all be 90 degrees from the zone axis on the stereographic projection. The zone axes were located by the following procedure:

(a) Place the stereographic projection on a Wulff net and rotate it until all ao the spots of one zone lie on a great circle.

(b) Measure 90 degrees along the equator of the Wulff net and mark the location of the zone axis on the projection.

(c) Repeat (a) and (b) for all zones

4. Measurement of angles between zone axes

The next step is to measure all of the angles between zone axes. This is done by rotating the stereographic projection on the Wulff net until each pair of zone axes fall on a great circle. Then the angle between them is measured along the great circle. This is repeated until the angle between every two pairs of zone axes has been measured. The angles measured for the spinel are shown in table 1.

5. Identification of the zone axes

Since the angles between all the zone axes in an isometric crystal can be calculated, the measured angles can be compared with them and the zone axes can be identified. In an isometric crystal the angles between the zone axes are equal to interplanar angles so a table of interplanar angles can be used (see table 3). All but one of the angles measured between the zone axes of the spinel agree within one degree of the calculated values. This can be seen by comparing tables 1&2.

6. Location of major zones and symmetry elements on the stereographic projection

The location of the symmetry elements helps to identify important zones. With the spinel it was done in the following steps:

MEASURED ANGLES

	A	B	C	D	E
A	-	25	29	40	29
B	-	-	26	20	40
C	-	-	-	19	19
D	-	-	-	-	38
E	-	-	-	-	-

A=211
B=310
C=110
D=321
E=321

TABLE 1

CALCULATED ANGLES

	A	B	C	D	E
A	-	25.4	30	40.2	29.2
B	-	-	26.6	21.6	40.5
C	-	-	-	19.1	19.1
D	-	-	-	-	38.2
E	-	-	-	-	-

TABLE 2

$((h_1k_1l_1))$	$((h_2k_2l_2))$	Possible angles (in degrees) between pairs of planes $(h_1k_1l_1)$ and $(h_2k_2l_2)$
100	100	0.00 90.00
	110	45.00 90.00
	111	54.74
	210	26.56 63.43 90.00
	211	35.26 65.90
	221	48.19 70.53
	310	18.43 71.56 90.00
	311	25.24 72.45
	320	33.69 56.31 90.00
	321	36.70 57.69 74.50
	322	43.31 60.98
	331	46.51 76.74
	332	50.24 64.76
	410	14.04 75.96 90.00
110	110	0.00 60.00 90.00
	111	35.26 90.00
	210	18.43 50.77 71.56
	211	30.00 54.74 73.22 90.00
	221	19.47 45.00 76.37 90.00
	310	26.56 47.87 63.43 77.08
	311	31.48 64.76 90.00
	320	11.31 53.96 66.91 78.69
	321	19.11 40.89 55.46 67.79 79.11
	322	30.96 46.69 80.12 90.00
	331	13.26 49.54 71.07 90.00
	332	25.24 41.08 81.33 90.00
	410	30.96 46.69 59.04 80.12
111	111	0.00 70.53
	210	39.23 75.04
	211	19.47 61.87 90.00
	221	15.79 54.74 78.90
	310	43.09 68.58
	311	29.50 58.52 79.98
	320	36.81 80.78
	321	22.21 51.89 72.02 90.00
	322	11.42 65.16 81.95
	331	22.00 48.53 82.39
	332	10.02 60.50 75.75
	410	45.56 65.16

$((h_1k_1l_1))$	$((h_2k_2l_2))$	Possible angles (in degrees) between pairs of planes $(h_1k_1l_1)$ and $(h_2k_2l_2)$
210	210	0.00 36.87 53.13 66.42 78.46 90.00
	211	24.09 43.09 56.79 79.48 90.00
	221	26.56 41.81 53.40 63.43 72.65 90.00
	310	8.13 31.95 45.00 64.90 73.57 81.87
	311	19.29 47.61 66.14 82.25
	320	7.12 29.74 41.91 60.25 68.15 75.64 82.87
	321	17.02 33.21 53.30 61.44 68.99 83.14 90.00
	322	29.80 40.60 49.40 64.29 77.47 83.77
	331	22.57 44.10 59.14 72.07 84.11
	332	30.89 40.29 48.13 67.58 73.38 84.53
	410	12.53 29.80 40.60 49.40 64.29 77.47 83.77
211	211	0.00 33.56 48.19 60.00 70.53 80.40
	221	17.72 35.26 47.12 65.90 74.21 82.18
	310	25.35 40.21 58.91 75.04 82.58
	311	10.02 42.39 60.50 75.75 90.00
	320	25.06 37.57 55.52 63.07 83.50
	321	10.89 29.20 40.20 49.11 56.94 70.89 77.40
		83.74 90.00
	322	8.05 26.98 53.55 60.32 72.72 78.58 84.32
	331	20.51 41.47 68.00 79.20
	332	16.78 29.50 52.46 64.20 69.62 79.98 85.01
	410	26.98 46.12 53.55 60.32 72.72 78.58
221	221	0.00 27.27 38.94 63.61 83.62 90.00
	310	32.51 42.45 58.19 65.06 83.95
	311	25.24 45.29 59.83 72.45 84.23
	320	22.41 42.30 49.67 68.30 79.34 84.70
	321	11.49 27.02 36.70 57.69 63.55 74.50 79.74
		84.89
	322	14.04 27.21 49.70 66.16 71.13 75.96 90.00
	331	6.21 32.73 57.64 67.52 85.61
	332	5.77 22.50 44.71 60.17 69.19 81.83 85.92
	410	36.06 43.31 55.53 60.98 80.69
310	310	0.00 25.84 36.87 53.13 72.54 84.26
	311	17.55 40.29 55.10 67.58 79.01 90.00
	320	15.26 37.87 52.12 58.25 74.74 79.90
	321	21.62 32.31 40.48 47.46 53.73 59.53 65.00
		75.31 85.15 90.00

$((h_1k_1l_1))$	$((h_2k_2l_2))$	Possible angles (in degrees) between pairs of planes (i. and $(h_2k_2l_2)$
310	322	32.47 46.35 52.15 57.53 72.13 76.70
	331	29.47 43.49 54.52 64.20 90.00
	332	36.00 42.13 52.64 61.84 66.14 78.33
	410	4.40 23.02 32.47 57.53 72.13 76.70 85
311	311	0.00 35.10 50.48 62.96 84.78
	320	23.09 41.18 54.17 65.28 75.47 85.20
	321	14.76 36.31 49.86 61.09 71.20 80.72
	322	18.07 36.45 48.84 59.21 68.55 85.81
	331	25.94 40.46 51.50 61.04 69.76 78.02
	332	25.85 39.52 50.00 59.05 67.31 75.10 90
	410	18.07 36.45 59.21 68.55 77.33 85.81
320	320	0.00 22.62 46.19 62.51 67.38 72.08
	321	15.50 27.19 35.38 48.15 53.63 58.74 68
		72.75 77.15 85.75 90.00
	322	29.02 36.18 47.73 70.35 82.27 90.00
	331	17.36 45.58 55.06 63.55 79.00
	332	27.50 39.76 44.80 72.80 79.78 90.00
	410	19.65 36.18 42.27 47.73 57.44 70.35 78
		82.27
321	321	0.00 21.79 31.00 38.21 44.41 49.99 64
		69.07 73.40 85.90
	322	13.51 24.84 32.57 44.52 49.59 63.01 71
		78.79 82.55 86.28
	331	11.19 30.85 42.63 52.18 60.63 68.42 75
		82.96 90.00
	332	14.38 24.26 31.27 42.20 55.26 59.15 62
		73.45 80.16 83.46 86.73
	410	24.84 32.57 44.52 49.59 54.31 63.01 67
		71.09 82.55 86.28
322	322	0.00 19.75 58.03 61.93 76.39 86.63
	331	18.93 33.42 43.67 59.95 73.85 80.97 86
	332	10.74 21.45 55.33 68.78 71.92 87.04
	410	34.57 49.68 53.97 69.33 72.90

331	331	0.00 26.52 37.86 61.73 80.91 86.98
	332	11.98 28.31 38.50 54.06 72.93 84.39 90.0
	410	33.42 43.67 52.26 59.95 67.08 86.81
332	332	0.00 17.34 50.48 65.85 79.52 82.16
	410	39.14 43.62 55.33 58.86 62.26 75.02
410	410	0.00 19.75 28.07 61.93 76.39 86.63 90.0

from Azaroff

Table 3

(a) By looking at a stereogram for a cubic crystal it can be seen that there is a mirror plane perpendicular to [110]. The trace of the mirror plane was drawn 90 degrees from [110] on the projection
(b)Since there were no other simple relationships between mirror planes and the other zone axes another zone was located. This was done by taking the cross product of two of the zone axes(in this case [110] and [211]) to find another perpendicular to them. The cross product of the two resulted in a [111] zone. This zone was plotted on the projection 90 degrees from the great circle containing the two zone axes.
(c) This [111] plotted on the trace of the mirror plane perpendicular [110]. From a stereogram and a table of angles it can be seen that a [100] zone plots 54.7 degrees to one side of the [111] zone on the trace of the mirror plane. A [100] zone was then plotted and the angles it made with the other zone axes was checked to make sure that it was the correct orientation. One of the two possible orientations did not work.
(d) With the [100] on the projection the position were easily obtained by simply comparing the projection with the stereogram.
(e) Indices were assigned to the intersections of the mirrror planes by assigning [001] to the four-fold axis closest to the center of the projection and assigning the rest of the indices by comparison with the stereogram.

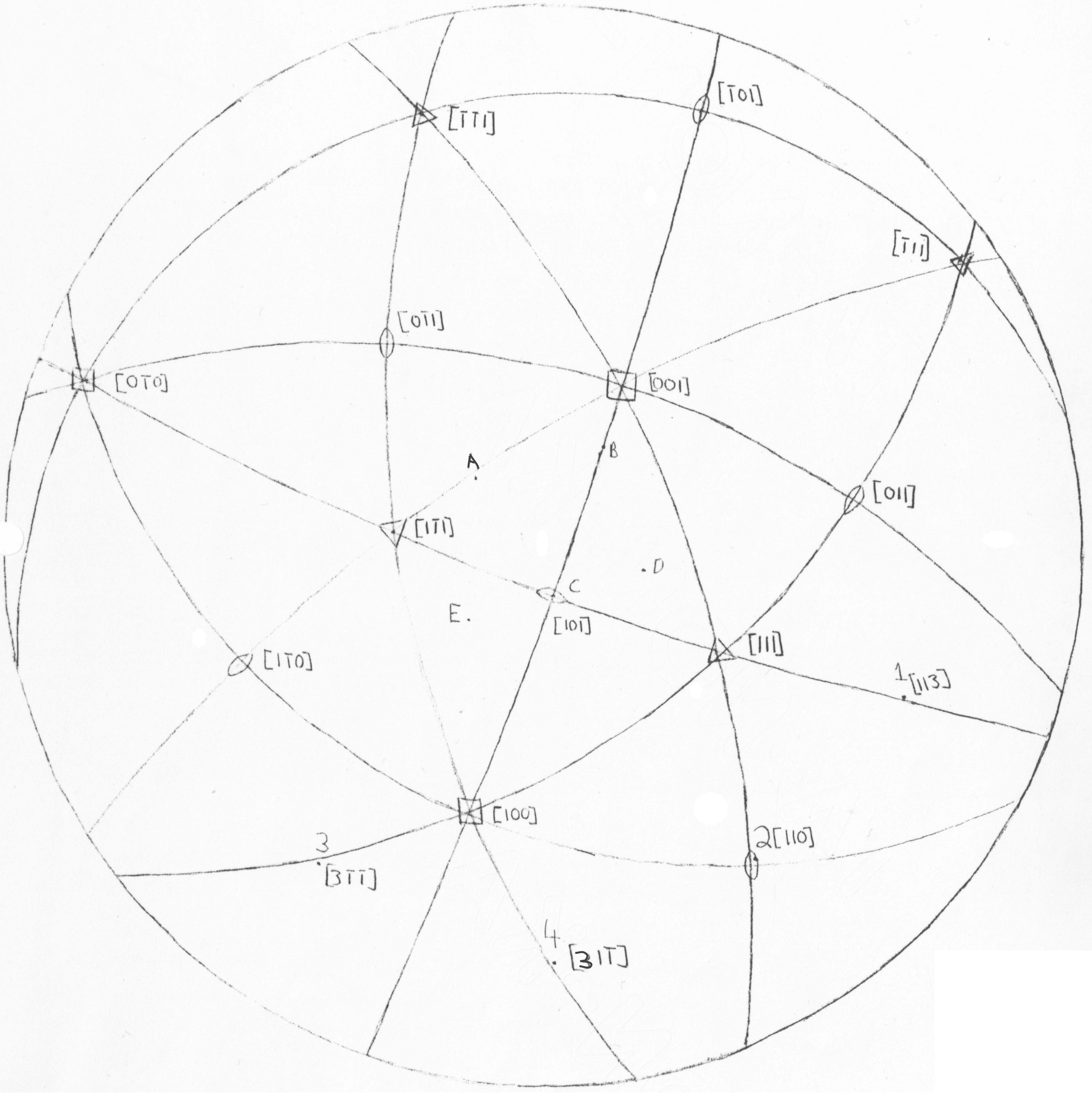
7. Determining indices of face poles

The indices of the poles labelled 1,2,3 and 4 on the stereographic projection were determined by using the formulas

$$h/k = \tan\phi \qquad (3)$$

$$l/k = [(\tan\rho)(\cos\phi)]^{-1} \qquad (4)$$

Set [001] to be $\rho=0$ and [010] to be $\rho=90$, $\phi=0$. To measure ρ for a pole rotate the stereographic projection on a Wulff net until the [001] and the being measured lie on a great circle. Measure the angle between them on the great circle. Measure ϕ by marking the intersection of the great circle containing the pole and [001] and the great circle containing [010] and [100]. The angle between [010] and the mark on the great circle containing



[$\bar{1}\bar{1}1$]
[$\bar{1}01$]
[$\bar{1}11$]
[$0\bar{1}1$]
[$0\bar{1}0$]
[001]
A
B
[011]
[$1\bar{1}1$]
D
C
[101]
E.
[111]
1 [113]
[$1\bar{1}0$]
[100]
2 [110]
3
[$3\bar{1}\bar{1}$]
4
[$31\bar{1}$]

[010] and [100] is the angle ϕ. The values obtained for ϕ and ρ were used in formulas (3) and (4) and the indices were determined by comparing the values obtained for h/k with l/k. The indices determined for the poles 1,2,3 and 4 were respectively: (113),(110), $(3\bar{1}\bar{1})$ and $(31\bar{1})$.

Conclusion

There was an excellent match between the measured and theoretical values of the angles between the zone axes. All but one of the angles between the zone axes was within one degre of these angles. This is accurate enough to give an unambiguous identification of the zone axes. From the stereographic projection (fig. 4) it is possible to determine the orientation of the crystal with respect to the x-ray beam. This method can be used for any isometric crystal in any orientation. Unfortunately this method is straightforward for isometric crystals because for nonisometric crystals it is necessary to know the relative lengths of the edges of the unit cell and the angles between them.

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